

## METHOD FOR SEPARATION AND RECOVERY OF AROMATIC COMPOUND AND HYDROGEN CHLORIDE

**Publication number:** JP2007008898 (A)

**Publication date:** 2007-01-18

**Inventor(s):** SEO TAKEO; SUZUTA TETSUYA +

**Applicant(s):** SUMITOMO CHEMICAL CO +

**Classification:**

- international: C01B7/01; C01B7/04; C07C15/04; C07C7/08; C01B7/00; C07C15/00; C07C7/00

- European:

**Application number:** JP20050194692 20050704

**Priority number(s):** JP20050194692 20050704

**Abstract of JP 2007008898 (A)**

**PROBLEM TO BE SOLVED:** To provide a method for separately recovering an aromatic compound and hydrogen chloride from a hydrogen chloride gas containing the aromatic compound. ; **SOLUTION:**

The separation and recovery method contains a chlorination step to react an aromatic compound with chlorine to obtain a chlorinated aromatic compound and hydrogen chloride; a hydrogen chloride purifying step to dissolve the hydrogen chloride gas containing the aromatic compound in a chlorinated aromatic compound to obtain a gas composed mainly of hydrogen chloride and a solution containing hydrogen chloride, the aromatic compound and the chlorinated aromatic compound; a hydrogen chloride recovering step to separate and recover the gas composed mainly of hydrogen chloride from the solution containing hydrogen chloride, the aromatic compound and the chlorinated aromatic compound; and an aromatic compound recovering step to separate and recover a fraction composed mainly of the aromatic compound from the fraction composed mainly of the aromatic compound and the chlorinated aromatic compound. ; **COPYRIGHT:** (C)2007,JPO&INPIT

L1 ANSWER 3 OF 5 HCAPLUS COPYRIGHT 2010 ACS on STN

AN 2007:54447 HCAPLUS Full-text

DN 146:124038

TI Method for separation and recovery of hydrogen chloride and aromatic

compounds from their mixtures obtained by chlorination of aromatic compounds

IN Seo, Takeo; Suzuta, Tetsuya

PA Sumitomo Chemical Co., Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 9pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
-----	----	-----	-----	----

PI JP 2007008898	A	20070118	JP 2005-194692	
------------------	---	----------	----------------	--

20050704 <--

PRAI JP 2005-194692 20050704

AB The method comprises the steps of: (1) chlorination of aromatic compds. (Ar) with Cl for manufacturing chlorinated aromatic compds. (CAr) and HCl (I) , (2) dissoln. of I gas containing unreacted Ar in CAr for obtaining I gas, and solns. containing I, Ar, and CAr, (3) separation of the solns. into I gas and distillates containing Ar and CAr, and (4) recovery of Ar from the distillates. Preferably, the I gas obtained in the process 3 is fed to the process 2 or (5) oxidation of I gas with O for reproducing Cl, and Ar recovered in the process 4 is fed to the process 1. Thus, manufacture of chlorobenzene by chlorination of benzene with Cl, and recovery of HCl and benzene by the above method is exemplified.

L3 ANSWER 2 OF 5 WPIX COPYRIGHT 2010 THOMSON REUTERS on STN  
 AN 2007-145950 [200715] WPIX Full-text  
 DNC C2007-052619 [200715]  
 TI Separation and recovery of aromatic compound and hydrogen chloride  
 involves dissolving hydrogen chloride gas containing aromatic  
 compound in  
 chlorinated compound, and separating produced gas and fraction of  
 aromatic  
 compound  
 DC E14; E36; H02  
 IN SEO T; SUZUTA T  
 PA (SUMO-C) SUMITOMO CHEM CO LTD  
 CYC 1  
 PI JP 2007008898 A 20070118 (200715)\* JA 9[1]  
 <--  
 ADT JP 2007008898 A JP 2005-194692 20050704  
 PRAI JP 2005-194692 20050704  
 IPCI C01B0007-00 [I,C]; C01B0007-01 [I,A]; C01B0007-04 [I,A]; C07C0015-  
 00  
 [I,C]; C07C0015-04 [I,A]; C07C0007-00 [I,C]; C07C0007-08 [I,A]  
 FCL C01B0007-01 H; C01B0007-04 A; C07C0015-04; C07C0007-08  
 FTRM 4G041; 4H006; 4H006/AA02; 4H006/AD13; 4H006/BD33; 4H006/BD41;  
 4H006/BD52;  
 4H006/BD53; 4H006/BD60  
 AB JP 2007008898 A UPAB: 20070301  
 NOVELTY - An aromatic compound and chlorine are reacted to obtain  
 chlorinated aromatic compound and hydrogen chloride. A hydrogen  
 chloride gas containing an aromatic compound is dissolved in a  
 chlorinated aromatic compound to obtain gas comprising hydrogen  
 chloride and solution containing hydrogen chloride, aromatic  
 compound and chlorinated aromatic compound. Subsequently, the gas  
 containing hydrogen chloride is separated from the solution and  
 the fraction containing aromatic compound is separated from the  
 fraction containing aromatic compound and chlorinated aromatic  
 compound.  
 USE - For separating and recovering aromatic compound and  
 hydrogen chloride.  
 ADVANTAGE - The method enables to recover aromatic  
 compounds efficiently and produce high quality hydrogen chloride  
 gas.  
 DESCRIPTION OF DRAWINGS - The figure shows the removal of  
 aromatic compound and chlorinated aromatics. (Drawing includes  
 non-English language text)  
 Chlorination process (A)  
 Hydrogen chloride purification process (B)  
 Pump (C,F)  
 Hydrogen chloride collection process (D)  
 Aromatic compound collection process (E)  
 MC CPI: E10-H03C1; E11-Q01B; E31-B02D; H02-D

# PATENT ABSTRACTS OF JAPAN

(11)Publication number : **2007-008898**

(43)Date of publication of application : **18.01.2007**

---

(51)Int.Cl.	<i>C07C</i>	<i>7/08</i>	<i>(2006.01)</i>
	<i>C07C</i>	<i>15/04</i>	<i>(2006.01)</i>
	<i>C01B</i>	<i>7/04</i>	<i>(2006.01)</i>
	<i>C01B</i>	<i>7/01</i>	<i>(2006.01)</i>

---

(21)Application number : **2005-194692** (71)Applicant : **SUMITOMO CHEMICAL CO LTD**

(22)Date of filing : **04.07.2005** (72)Inventor : **SEO TAKEO  
SUZUTA TETSUYA**

---

## **(54) METHOD FOR SEPARATION AND RECOVERY OF AROMATIC COMPOUND AND HYDROGEN CHLORIDE**

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a method for separately recovering an aromatic compound and hydrogen chloride from a hydrogen chloride gas containing the aromatic compound.

SOLUTION: The separation and recovery method contains a chlorination step to react an aromatic compound with chlorine to obtain a chlorinated aromatic compound and hydrogen chloride; a hydrogen chloride purifying step to dissolve the hydrogen chloride gas containing the aromatic compound in a chlorinated aromatic compound to obtain a gas composed mainly of hydrogen chloride and a solution containing hydrogen chloride, the aromatic compound and the chlorinated aromatic compound; a hydrogen chloride recovering step to separate and recover the gas composed mainly of hydrogen chloride from the solution containing hydrogen chloride,

the aromatic compound and the chlorinated aromatic compound; and an aromatic compound recovering step to separate and recover a fraction composed mainly of the aromatic compound from the fraction composed mainly of the aromatic compound and the chlorinated aromatic compound.

(19) 日本国特許庁(JP)

(12) 公開特許公報(A)

(11) 特許出願公開番号

特開2007-8898

(P2007-8898A)

(43) 公開日 平成19年1月18日(2007.1.18)

(51) Int. Cl.

F 1

テーマコード (参考)

C07C 7/08 (2006.01)

C07C 7/08

4H006

C07C 15/04 (2006.01)

C07C 15/04

C01B 7/04 (2006.01)

C01B 7/04

A

C01B 7/01 (2006.01)

C01B 7/01

H

審査請求 未請求 請求項の数 5 O L (全 9 頁)

(21) 出願番号 特願2005-194692 (P2005-194692)

(22) 出願日 平成17年7月4日(2005.7.4)

(71) 出願人 000002093

住友化学株式会社

東京都中央区新川二丁目27番1号

(74) 代理人 100093285

弁理士 久保山 隆

(74) 代理人 100113000

弁理士 中山 亨

(74) 代理人 100119471

弁理士 榎本 雅之

(72) 発明者 瀬尾 健男

千葉県市原市姉崎海岸5の1 住友化学株式会社内

(72) 発明者 鈴田 哲也

愛媛県新居浜市惣開町5番1号 住友化学株式会社内

最終頁に続く

(54) 【発明の名称】 芳香族化合物と塩化水素の分離回収方法

(57) 【要約】

【課題】 芳香族化合物を含有する塩化水素のガスから芳香族化合物と塩化水素を各々分離して回収する。

【解決手段】 下記の工程を用いる。

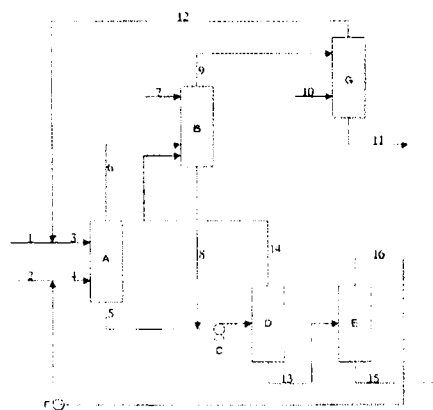
塩素化工程：芳香族化合物と塩素を反応させ、塩素化芳香族化合物と塩化水素を得る

塩化水素精製工程：芳香族化合物を含有する塩化水素ガスを塩素化芳香族化合物に溶解させて、塩化水素を主とするガスと、塩化水素、芳香族化合物及び塩素化芳香族化合物を含む溶液を得る

塩化水素回収工程：塩化水素、芳香族化合物、塩素化芳香族化合物を含む溶液から塩化水素を主とするガスを分離、回収する

芳香族化合物回収工程：芳香族化合物及び塩素化芳香族化合物を主とする留分から芳香族化合物を主とする留分を分離、回収する

【選択図】 図1



## 【特許請求の範囲】

## 【請求項1】

下記の塩素化工程で得られる芳香族化合物を含有する塩化水素のガスから芳香族化合物と塩化水素を各々分離して回収する方法であって、芳香族化合物を含有する塩化水素ガスを下記塩化水素精製工程に付すことにより塩素化芳香族化合物に溶解させて塩化水素、芳香族化合物及び塩素化芳香族化合物を含む溶液とし、該溶液を下記塩化水素回収工程に付すことにより塩化水素を主とするガスと芳香族化合物及び塩素化芳香族化合物を主とする留分を得、該芳香族化合物及び塩素化芳香族化合物を主とする留分を下記の芳香族化合物回収工程へ供給する塩化水素と芳香族化合物の分離回収方法。

塩素化工程：芳香族化合物と塩素を反応させ、塩素化芳香族化合物と塩化水素を得る工程

塩化水素精製工程：芳香族化合物を含有する塩化水素ガスを塩素化芳香族化合物に溶解させて、塩化水素を主とするガスと、塩化水素、芳香族化合物及び塩素化芳香族化合物を含む溶液を得る工程

塩化水素回収工程：塩化水素、芳香族化合物、塩素化芳香族化合物を含む溶液から塩化水素を主とするガスを分離、回収する工程

芳香族化合物回収工程：芳香族化合物及び塩素化芳香族化合物を主とする留分から、芳香族化合物を主とする留分を分離、回収する工程

## 【請求項2】

塩化水素回収工程で得られた塩化水素を主とするガスを塩化水素精製工程へ供給する請求項1記載の方法

## 【請求項3】

芳香族化合物回収工程で得られた芳香族化合物を主とする留分を塩素化工程へ供給する請求項1記載の方法。

## 【請求項4】

塩化水素精製工程で得られた塩化水素を主とするガスを下記の酸化工程へ供給する請求項1記載の方法。

酸化工程：塩化水素を酸素と反応させ、塩素を得る工程

## 【請求項5】

芳香族化合物がベンゼンであり、塩素化芳香族化合物がクロルベンゼンである請求項1記載の方法。

## 【発明の詳細な説明】

## 【技術分野】

## 【0001】

本発明は、芳香族化合物と塩化水素の分離回収方法に関するものである。更に詳しくは、本発明は、芳香族化合物と塩素を反応させることにより、塩素化芳香族化合物と塩化水素を得る工程（塩素化工程）で得られる芳香族化合物を含有する塩化水素のガスから芳香族化合物と塩化水素を各々分離して回収する方法であって、工業的に有用な高品位の塩化水素ガスを得ると同時に芳香族化合物を効率的に回収するという優れた特徴を有する芳香族化合物と塩化水素の分離回収方法に関するものである。

## 【背景技術】

## 【0002】

たとえばベンゼンのような芳香族化合物と塩素からクロルベンゼン（モノクロルベンゼンを意味する。以下、同じ。）のような塩素化芳香族化合物を製造する方法は公知である（たとえば、特許文献1参照。）。

## 【0003】

ところで、芳香族化合物と塩素の反応では芳香族化合物及び塩素化芳香族化合物が含まれる塩化水素のガスが副生する。塩化水素ガスは酸素と反応させて塩素を得、該塩素を芳香族化合物との反応にリサイクル使用する等の有効利用が可能である。塩化水素ガスを有効利用するにあたっては、その中に含まれる芳香族化合物と塩化水素を分離回収する必要

がある。

【0004】

【特許文献1】米国特許第2653904号明細書

【発明の開示】

【発明が解決しようとする課題】

【0005】

かかる状況において、本発明が解決しようとする課題は、芳香族化合物と塩素を反応させることにより、塩素化芳香族化合物と塩化水素を得る工程（塩素化工程）で得られる芳香族化合物を含有する塩化水素のガスから芳香族化合物と塩化水素を各々分離して回収する方法であって、工業的に有用な高品位の塩化水素ガスを得ると同時に芳香族化合物を効率的に回収するという優れた特徴を有する芳香族化合物と塩化水素の分離回収方法を提供する点にある。

【課題を解決するための手段】

【0006】

すなわち、本発明は、下記の塩素化工程で得られる芳香族化合物を含有する塩化水素のガスから芳香族化合物と塩化水素を各々分離して回収する方法であって、芳香族化合物を含有する塩化水素ガスを下記塩化水素精製工程に付すことにより塩素化芳香族化合物に溶解させて塩化水素、芳香族化合物及び塩素化芳香族化合物を含む溶液とし、該溶液を下記塩化水素回収工程に付すことにより塩化水素を主とするガスと芳香族化合物及び塩素化芳香族化合物を主とする留分を得、該芳香族化合物及び塩素化芳香族化合物を主とする留分を下記の芳香族化合物回収工程へ供給する塩化水素と芳香族化合物の分離回収方法に係るものである。

塩素化工程：芳香族化合物と塩素を反応させ、塩素化芳香族化合物と塩化水素を得る工程

塩化水素精製工程：芳香族化合物を含有する塩化水素ガスを塩素化芳香族化合物に溶解させて、塩化水素を主とするガスと、塩化水素、芳香族化合物及び塩素化芳香族化合物を含む溶液を得る工程

塩化水素回収工程：塩化水素、芳香族化合物、塩素化芳香族化合物を含む溶液から塩化水素を主とするガスを分離、回収する工程

芳香族化合物回収工程：芳香族化合物及び塩素化芳香族化合物を主とする留分から芳香族化合物を主とする留分を分離、回収する工程

【発明の効果】

【0007】

本発明により、芳香族化合物と塩素を反応させることにより、塩素化芳香族化合物と塩化水素を得る工程（塩素化工程）で得られる芳香族化合物を含有する塩化水素のガスから芳香族化合物と塩化水素を各々分離して回収する方法であって、工業的に有用な高品位の塩化水素ガスを得ると同時に芳香族化合物を効率的に回収するという優れた特徴を有する芳香族化合物と塩化水素の分離回収方法を提供することができる。

【発明を実施するための最良の形態】

【0008】

本発明は、下記の塩素化工程で得られる芳香族化合物を含有する塩化水素のガスから芳香族化合物と塩化水素を各々分離して回収する方法である。

塩素化工程：芳香族化合物と塩素を反応させ、塩素化芳香族化合物と塩化水素を得る工程

【0009】

芳香族化合物としては、ベンゼン、トルエン、キシレン等を上げることができる。塩素化芳香族化合物としては、クロルベンゼン等をあげることができる。芳香族化合物としてベンゼンを用い、塩素化芳香族化合物としてクロルベンゼンを得る方法が産業上の観点から特に重要である。

【0010】

芳香族化合物と塩素を反応させる方法については、特に制限はなく、公知の方法を使用することができる。具体的な方法の例を示すと、次のとおりである。芳香族化合物と塩素のモル比（芳香族化合物／塩素）は3以上であり、反応温度は25～140℃であり、反応圧力は0.02～1.0MPaである。反応器としては、たとえば槽型反応器を用いることができる。反応には触媒としてFeCl<sub>2</sub>を用いることができる。

【0011】

塩素化工程では副反応物として塩化水素が発生する。該塩化水素のガスを回収するには、たとえば反応器内に液相部と気相部を設け、塩素を液相に吹き込んで芳香族化合物と反応させ塩素化芳香族化合物を得、副生する塩化水素ガスを気相から抜き出せばよい。抜き出した塩化水素ガスは直接塩化水素精製工程に供給することもできるが、塩素化反応温度が常温より高い場合、塩素化精製工程までの間に熱交換器を設けて冷却することによりガス中の芳香族化合物の一部を凝縮、分離して含有量を低減させた後、塩化水素精製工程に供給することもできる。

【0012】

塩素化工程から回収される塩化水素のガス中には、未反応の芳香族化合物が含まれる。

【0013】

本発明においては、上記の芳香族化合物を含有する塩化水素ガスを塩素化芳香族化合物に溶解させて塩化水素、芳香族化合物及び塩素化芳香族化合物を含む溶液とする。

【0014】

溶液を得る方法としては、充填塔あるいは棚段塔の下部より芳香族化合物を含む塩化水素ガスを供給し、上部より塩素化芳香族化合物を主として含む溶液を供給し向流接触させる塔方式、芳香族化合物を含む塩化水素ガスを管塔の上部から塩素化芳香族を主とする液と共に流下させて並流接触させ、下部で気液分離する濡れ壁方式などを例示することができる。

【0015】

塩素化芳香族化合物としては、クロルベンゼン、ジクロルベンゼン等を挙げることができる。

【0016】

本発明においては、上記の溶液を塩化水素回収工程に付すことにより塩化水素を主とするガスと芳香族化合物及び塩素化芳香族化合物を主とする留分を得る。

【0017】

塩化水素回収工程を実施する方法としては、たとえば塩化水素精製工程で得た溶液を、底部にリボイラーを備える蒸留塔に供給し、リボイラーで塔底液を加熱して溶解する塩化水素を放散させればよい。塔底から得られる芳香族化合物及び塩素化芳香族化合物を主とする留分中の塩化水素のモル濃度は0.5%以下が好ましく、更に好ましくは1～1000ppmである。該濃度が高すぎると、次の芳香族化合物回収工程で回収される芳香族化合物に混入した塩化水素による塔頂での凝縮温度低下などの問題を引き起こすことがある。一方1ppm以下にするのは大きな設備やエネルギーが必要になり経済的でない。蒸留塔の操作圧力は減圧、常圧、加圧いずれでもよいが、該圧力を塩化水素精製工程の圧力より高く保ち、塩素化精製工程で得た液をポンプを用いて塩化水素回収工程に送入することで、蒸留塔の塔頂から得た塩化水素ガスを圧縮することなく塩化水素精製工程に供給し、精製することができる。この際、塔頂から得たガスを一旦熱交換器で冷却し、ガスに含まれる芳香族化合物及び塩素化芳香族化合物の一部を凝縮させて塔に還流させることもできる。

【0018】

本発明においては、上記の塩化水素回収工程で得られた芳香族化合物及び塩素化芳香族化合物を主とする留分を下記の芳香族化合物回収工程へ供給する。

芳香族化合物回収工程：芳香族化合物及び塩素化芳香族を主とする留分から、芳香族化合物を主とする留分を分離、回収する工程

【0019】



芳香族化合物回収工程を実施する具体例をあげると次のとおりである。

【0020】

頂部にコンデンサー、底部にリボイラーを備える蒸留塔の中段部に塩化水素回収工程で得られた芳香族化合物及び塩素化芳香族化合物を主とする留分を供給する。リボイラーでの加熱により塔頂から得られる蒸気をコンデンサーで液化し、その一部を蒸留塔の頂部に還流させることにより、塔頂から芳香族化合物に富む留分が得られる。蒸留塔には塩化水素回収工程で得られた芳香族化合物および塩素化芳香族化合物を主とする留分のみを供給してもよいが、たとえば塩素化工程から得られる、未反応の芳香族化合物と反応で生成した塩素化芳香族からなる液を併せて供給し、芳香族化合物を回収することもできる。本発明においては、芳香族化合物回収工程で得られた芳香族化合物を主とする留分を塩素化工程へ供給することができる。このことにより芳香族化合物を有用にリサイクル利用できる。

【0021】

本発明においては、塩化水素精製工程で得られた塩化水素を主とするガスを下記の酸化工程へ供給することができる。このことにより、塩化水素を塩素に変換し、該塩素を有効利用することができる。

酸化工程：塩化水素を酸素と反応させ、塩素を得る工程

【0022】

酸化工程は、塩素化工程で得た塩化水素を酸素と反応させ、塩素を得る工程である。塩化水素と酸素を反応させる方法については、特に制限はなく、公知の方法を使用することができる。具体的な方法の例を示すと、次のとおりである。塩化水素と酸素のモル比（塩化水素／酸素）は0.5～2であり、反応温度は200～500℃、好ましくは200～380℃であり、反応圧力は0.1～5MPaであり、空塔速度は0.7～10m/sである。反応器としては、固定床反応器、流動床反応器、移動床反応器を用いることができる。反応には触媒として酸化クロム触媒、酸化ルテニウム触媒を用いることができる。

【0023】

酸化工程で得られた塩素の少なくとも一部は塩素化工程へリサイクルされ、塩素化工程の原料として有効利用される。

【0024】

上記の態様を採用することにより、芳香族炭化水素（たとえば、ベンゼン）と塩素とから最終目的物としての塩素化芳香族炭化水素（たとえば、クロルベンゼン）を効率的に製造することができる。

【実施例】

【0025】

次に本発明を実施例により説明する。

実施例1

本発明はたとえば図1のフローと表1の物質収支により最適に実施することができる。

ベンゼンと塩素を反応させてクロルベンゼンと塩化水素を得た。ここで反応温度は117℃、反応圧力は0.6MPaとし、外部から供給されるベンゼン／塩素のモル比は8／1とした。反応で発生したベンゼンと塩化水素を含むガスは30℃まで冷却して凝縮したベンゼンは戻し、ベンゼンと塩化水素を0.03／1で含む混合ガス（流体番号－6）を取り出した。〔塩素化工程〕

上記の混合ガスとクロルベンゼンを圧力0.55MPa、温度が－35℃の下で接触させ、ベンゼン、クロルベンゼンの濃度が低減された塩化水素ガス（流体番号－9）と、塩化水素水素、ベンゼン、クロルベンゼンからなる溶液（流体番号－8）を得た。〔塩化水素精製工程〕

塩化水素精製工程で得られた溶液を、塩素化反応で得られた塩化水素、ベンゼン、クロルベンゼンを含む液と共に圧力0.6MPa下で加熱して液中に溶解している塩化水素を放散させ、塔頂からベンゼンと塩化水素を0.02／1で含む混合ガス（流体番号－14）を、塔底から塩化水素のモル濃度が低減されたベンゼンおよびクロルベンゼンの混合液

( 流体番号－１３ ) を得た。〔塩化水素回収工程〕

塩化水素回収工程で得られた混合ガスは塩化水素精製工程に戻し、再精製した。塩化水素回収工程から得られたベンゼンおよびクロルベンゼンの混合液は通常の蒸留によりベンゼン ( 流体番号－１６ ) とクロルベンゼン ( 流体番号－１５ ) に分離した。〔芳香族化合物回収工程〕

芳香族化合物回収工程で分離、回収されたベンゼンは塩素化工程の原料としてリサイクルした。また、塩化水素精製工程で得られた塩化水素ガスは酸素と反応させ、塩素と水に変換した。〔酸化工程〕

得られた塩素 ( 流体番号－１２ ) は塩素化工程の原料としてリサイクルした。

【表1】

流体番号	1	2	3	4	5	6	7	8
温度 (°C)	25	25	38	108	117	30	25	-35
圧力 (MPa)	0.65	0.65	0.65	0.65	0.60	0.60	0.06	0.55
モル流量 kg-mol/hr								
塩素	221.0		421.0					
塩化水素				0.3	104.4	316.9		70.6
ベンゼン		421.4		3368.0	2941.6	8.3		12.2
モノクロルベンゼン					415.1	0.1	30.0	30.2
酸素								
水					2.9			
他								
計	221.0	421.4	421.0	3368.3	3464.0	325.3	37.7	134.5
モル濃度 (%)								
塩素	100.0		100.0					
塩化水素				100ppm	1.7	97.4		62.5
ベンゼン		100.0		100.0	86.0	2.6		9.4
モノクロルベンゼン					12.1	150ppm	100.0	28.1
酸素								
水								
他					0.1			
計	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

流体番号	9	10	11	12	13	14	15	16
温度 (°C)	-35	25	25	25	157	30	168	25
圧力 (MPa)	0.55	0.50	0.50	0.50	0.60	0.60	0.25	0.25
モル流量 kg-mol/hr								
塩素				200.0				
塩化水素	421.0		21.0		0.3	174.7		0.3
ベンゼン					2949.9	3.9	3.3	2946.7
モノクロルベンゼン					445.1	0.1	445.1	
酸素		100.0						
水			200.0					
他				0.0	2.9		2.9	
計	421.0	100.0	221.1	200.0	3398.3	178.7	451.3	2946.9
モル濃度 (%)								
塩素				100.0				
塩化水素	100.0		9.5		100ppm	97.7		120ppm
ベンゼン	80ppm				86.8	2.2	0.7	100.0
モノクロルベンゼン	15ppm				13.1	0.1	98.6	
酸素		100.0						
水			90.5					
他					0.1		0.6	
計	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

【図面の簡単な説明】

【0027】

【図1】本発明を実施するフローの例である。

【符号の説明】

【0028】

A：塩素化工程

B：塩化水素精製工程

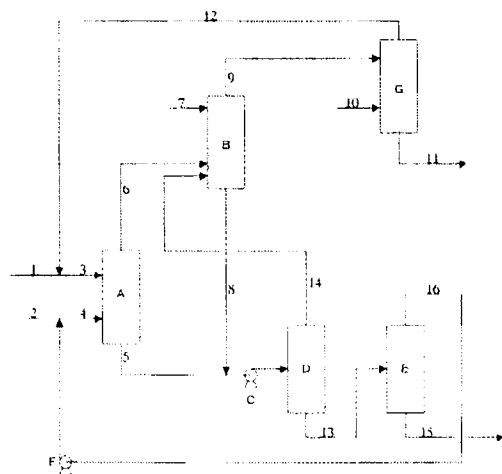
C、F：ポンプ

D：塩化水素回収工程

E：芳香族化合物回収工程

G：酸化工程

【図1】



F ターム(参考) 4H006 AA02 AD13 BD33 BD41 BD52 BD53 BD60

## Machine translation JP2007008898

### DETAILED DESCRIPTION

(19)**Publication country**Japan Patent Office (JP)  
(12)**Kind of official gazette**Publication of patent applications (A)  
(11)**Publication No.**JP,2007-8898,A (P2007-8898A)  
(43)**Date of Publication**January 18, Heisei 19 (2007.1.18)  
(54)**Title of the Invention**A separate recovery method of aromatic compounds and hydrogen chloride  
(51)**International Patent Classification**

C07C 7/08 (2006.01)

C07C 15/04 (2006.01)

C01B 7/04 (2006.01)

C01B 7/01 (2006.01)

**FI**

C07C 7/08

C07C 15/04

C01B 7/04 A

C01B 7/01 H

**Request for Examination**Unrequested

**The number of claims** 5

**Mode of Application**OL

**Number of Pages**9

(21)**Application number**Application for patent 2005-194692 (P2005-194692)

(22)**Filing date**July 4, Heisei 17 (2005.7.4)

(71)**Applicant**

**Identification Number**000002093

**Name**Sumitomo Chemical Co., Ltd.

**Address**2-27-1, Shinkawa, Chuo-ku, Tokyo

(74)**Attorney**

**Identification Number**100093285

**Patent Attorney**

**Name**Takashi Kuboyama

(74)**Attorney**

**Identification Number**100113000

**Patent Attorney**

**Name**Nakayama \*\*

(74)**Attorney**

**Identification Number**100119471

**Patent Attorney**

**Name**Masayuki Enomoto

(72)**Inventor(s)**

**Name**Takeo Senoo

**Address**5-1, Anesaki Kaigan, Ichihara-shi, Chiba-ken Inside of Sumitomo Chemical Co., Ltd.

(72)**Inventor(s)**

**Name**Tetsuya Suzuta

**Address** 5-1, Sobiraki-cho, Niihama-shi, Ehime-ken Inside of Sumitomo Chemical Co., Ltd.  
**Theme code (reference)**

4H006

**F-term (reference)**

4H006 AA02 AD13 BD33 BD41 BD52 BD53 BD60

---

**(57)Abstract**

**Technical problem** It dissociates respectively and aromatic compounds and hydrogen chloride are collected from the gas of hydrogen chloride containing aromatic compounds.

**Means for Solution** The following process is used.

Chlorination process: Make chlorine react to aromatic compounds and obtain chlorinated aromatics and hydrogen chloride.

Hydrogen-chloride purification process: Dissolve hydrogen chloride gas containing aromatic compounds in chlorinated aromatics, and obtain gas which is mainly concerned with hydrogen chloride, and a solution containing hydrogen chloride, aromatic compounds, and chlorinated aromatics.

Hydrogen chloride recovering process: Separate gas with which it is mainly concerned, and collect hydrogen chloride from a solution containing hydrogen chloride, aromatic compounds, and chlorinated aromatics.

Aromatic-compounds recovery process: Separate a fraction with which it is mainly concerned, and collect aromatic compounds from a fraction which is mainly concerned with aromatic compounds and chlorinated aromatics.

**Chosen drawing** Drawing 1

---

**Claim(s)**

**Claim 1**

It is the method of dissociating respectively and collecting aromatic compounds and hydrogen chloride from gas of hydrogen chloride containing aromatic compounds obtained at the following chlorination process, By giving hydrogen chloride gas containing aromatic compounds to the following hydrogen chloride purification process, make it dissolve in chlorinated aromatics and Hydrogen chloride, Consider it as a solution containing aromatic compounds and chlorinated aromatics, and a fraction which is mainly concerned with gas, aromatic compounds, and chlorinated aromatics which are mainly concerned with hydrogen chloride by giving this solution to the following hydrogen chloride recovering process is obtained, A separate recovery method of hydrogen chloride and aromatic compounds which supplies a fraction which is mainly concerned with these aromatic compounds and chlorinated aromatics to the following aromatic-compounds recovery process.

A chlorination process: A process of making chlorine reacting to aromatic compounds and obtaining chlorinated aromatics and hydrogen chloride

A process characterized by comprising the following of obtaining a solution.

A hydrogen-chloride purification process: Gas which dissolves hydrogen chloride gas containing aromatic compounds in chlorinated aromatics, and is mainly concerned with hydrogen chloride. Hydrogen chloride, aromatic compounds, and chlorinated aromatics.

A hydrogen chloride recovering process: A process which separates gas which is mainly concerned with hydrogen chloride, and are collected from a solution containing hydrogen chloride, aromatic compounds, and chlorinated aromatics

An aromatic-compounds recovery process: A process of separating a fraction with which it is mainly concerned, and collecting aromatic compounds from a fraction which is mainly concerned with aromatic compounds and chlorinated aromatics

**Claim 2**

A method according to claim 1 of supplying gas which is mainly concerned with hydrogen chloride obtained by a hydrogen chloride recovering process to a hydrogen chloride purification process

**Claim 3**

A method according to claim 1 of supplying a fraction which is mainly concerned with aromatic compounds obtained by an aromatic-compounds recovery process to a chlorination process.

**Claim 4**

A method according to claim 1 of supplying gas which is mainly concerned with hydrogen chloride obtained by a hydrogen chloride purification process to the following oxidation process.

An oxidation process: A process of making hydrogen chloride reacting to oxygen and obtaining chlorine

**Claim 5**

A way according to claim 1 aromatic compounds are benzene and chlorinated aromatics are chlorobenzene.

---

**Detailed Description of the Invention****Field of the Invention****0001**

This invention relates to the separate recovery method of aromatic compounds and hydrogen chloride. Chlorine is made to react to aromatic compounds by this invention in detail. Therefore, it is the method of dissociating respectively and collecting aromatic compounds and hydrogen chloride from the gas of hydrogen chloride containing chlorinated aromatics and the aromatic compounds obtained at the process (chlorination process) of obtaining hydrogen chloride, It is related with the separate recovery method of aromatic compounds and hydrogen chloride of having the outstanding feature of collecting aromatic compounds efficiently at the same time it obtains useful high-definition hydrogen chloride gas industrially.

**Background of the Invention****0002**

From aromatic compounds like benzene, and chlorine to for example, chlorobenzene (mono-chlorobenzene is meant.) Hereafter, it is the same. The method of manufacturing chlorinated aromatics **like** is publicly known (for example, refer to patent documents 1.).

**0003**

By the way, at the reaction of aromatic compounds and chlorine, the gas of hydrogen chloride by which aromatic compounds and chlorinated aromatics are contained carries out a byproduction. Effective use of making it react to oxygen, obtaining chlorine, and carrying out recycle employment of this chlorine to a reaction with aromatic compounds is possible for hydrogen chloride gas. In using hydrogen chloride gas effectively, it is necessary to carry out separate recovery of the aromatic compounds and hydrogen chloride which are contained in it.

**0004**

**Patent documents 1** U.S. Pat. No. 2653904 specification

**Description of the Invention****Problem(s) to be Solved by the Invention****0005**

In this situation, the issue which this invention tends to solve, It is the method of dissociating respectively and collecting aromatic compounds and hydrogen chloride from the gas of the hydrogen chloride which contains chlorinated aromatics and the aromatic compounds obtained at the process (chlorination process) of obtaining hydrogen chloride by making chlorine reacting to aromatic compounds, It is in the aromatic compounds which have the outstanding feature of collecting aromatic compounds efficiently, and the point of providing the separate recovery method of hydrogen chloride at the same time it obtains useful high-definition hydrogen chloride gas industrially.

**Means for Solving the Problem****0006**



Namely, this invention is the method of dissociating respectively and collecting aromatic compounds and hydrogen chloride from gas of hydrogen chloride containing aromatic compounds obtained at the following chlorination process, By giving hydrogen chloride gas containing aromatic compounds to the following hydrogen chloride purification process, make it dissolve in chlorinated aromatics and Hydrogen chloride, Consider it as a solution containing aromatic compounds and chlorinated aromatics, and a fraction which is mainly concerned with gas, aromatic compounds, and chlorinated aromatics which are mainly concerned with hydrogen chloride by giving this solution to the following hydrogen chloride recovering process is obtained, A separate recovery method of hydrogen chloride and aromatic compounds which supplies a fraction which is mainly concerned with these aromatic compounds and chlorinated aromatics to the following aromatic-compounds recovery process is started.

Chlorination process: A process of making chlorine reacting to aromatic compounds and obtaining chlorinated aromatics and hydrogen chloride

Hydrogen-chloride purification process: A process of obtaining gas which dissolves hydrogen chloride gas containing aromatic compounds in chlorinated aromatics, and is mainly concerned with hydrogen chloride, and a solution containing hydrogen chloride, aromatic compounds, and chlorinated aromatics

Hydrogen chloride recovering process: A process which separates gas which is mainly concerned with hydrogen chloride, and are collected from a solution containing hydrogen chloride, aromatic compounds, and chlorinated aromatics

Aromatic-compounds recovery process: A process of separating a fraction with which it is mainly concerned, and collecting aromatic compounds from a fraction which is mainly concerned with aromatic compounds and chlorinated aromatics

#### **Effect of the Invention**

##### **0007**

It is the method of dissociating respectively and collecting aromatic compounds and hydrogen chloride from the gas of hydrogen chloride containing the aromatic compounds obtained at the process (chlorination process) of obtaining chlorinated aromatics and hydrogen chloride by making chlorine reacting to aromatic compounds by this invention, While obtaining useful high-definition hydrogen chloride gas industrially, the separate recovery method of aromatic compounds and hydrogen chloride of having the outstanding feature of collecting aromatic compounds efficiently can be provided.

#### **Best Mode of Carrying Out the Invention**

##### **0008**

This invention is the method of dissociating respectively and collecting aromatic compounds and hydrogen chloride from the gas of hydrogen chloride containing the aromatic compounds obtained at the following chlorination process.

Chlorination process: The process of making chlorine reacting to aromatic compounds and obtaining chlorinated aromatics and hydrogen chloride

##### **0009**

Benzene, toluene, xylene, etc. can be raised as aromatic compounds. Chlorobenzene etc. can be raised as chlorinated aromatics. The method of obtaining chlorobenzene as chlorinated aromatics, using benzene as aromatic compounds is important especially from an industrial viewpoint.

##### **0010**

About the method of making chlorine reacting to aromatic compounds, there is no restriction in particular and a publicly known method can be used. It is as follows when the example of a concrete method is shown. The mole ratio (aromatic compounds/chlorine) of aromatic compounds and chlorine is three or more, reaction temperature is 25-140 \*\*, and reaction pressure is 0.02 - 1.0MPa. As a reactor, a tank reactor can be used, for example.  $\text{FeCl}_2$  can be used for a reaction as a catalyst.

##### **0011**

At a chlorination process, hydrogen chloride occurs as a side reaction thing. Provide a liquid phase part and a gas phase portion, for example in a reactor, blow chlorine into the liquid phase, it is made to react to aromatic compounds, and what is necessary is just to extract the hydrogen chloride gas which obtains and carries out the byproduction of the chlorinated aromatics from the gaseous phase, in order to collect the gas of this hydrogen chloride. Although extracted hydrogen

chloride gas can also be directly supplied to a hydrogen chloride purification process, When chlorinating reaction temperature is higher than ordinary temperature, after condensing some aromatic compounds in gas, dissociating and reducing content by providing a heat exchanger in before a chlorination purification process, and cooling, a hydrogen chloride purification process can also be supplied.

#### **0012**

Unreacted aromatic compounds are contained in the gas of the hydrogen chloride collected from a chlorination process.

#### **0013**

In this invention, it is considered as the solution which dissolves the hydrogen chloride gas containing the above-mentioned aromatic compounds in chlorinated aromatics, and contains hydrogen chloride, aromatic compounds, and chlorinated aromatics.

#### **0014**

As a method of obtaining a solution, the hydrogen chloride gas which contains aromatic compounds from the lower part of a packed column or a tray tower is supplied, it is made to flow down with the liquid which is mainly concerned with the upper part of \*\*\*\* to chlorination aromatic series, parallel flow contact is carried out, and vapor liquid separation of the tower method to which countercurrent contact of the solution which mainly contains chlorinated aromatics is supplied and carried out from the upper part, and the hydrogen chloride gas containing aromatic compounds is carried out in the lower part -- it gets wet and a wall method etc. can be illustrated.

#### **0015**

Chlorobenzene, dichlorobenzene, etc. can be mentioned as chlorinated aromatics.

#### **0016**

In this invention, the fraction which is mainly concerned with the gas, aromatic compounds, and chlorinated aromatics which are mainly concerned with hydrogen chloride is obtained by giving the above-mentioned solution to a hydrogen chloride recovering process.

#### **0017**

What is necessary is to supply the solution obtained, for example by the hydrogen chloride purification process as a method of carrying out a hydrogen chloride recovering process to the distillation column which equips a pars basilaris ossis occipitalis with a reboiler, and just to make the hydrogen chloride which heats a bottom and dissolves with a reboiler diffuse. 0.5% or less is desirable still more preferred, and the molar concentration of hydrogen chloride in the fraction which is mainly concerned with the aromatic compounds and chlorinated aromatics which are obtained from a bottom is 1-1000 ppm. When this concentration is too high, problems, such as a condensation temperature fall in the overhead by the hydrogen chloride mixed in the aromatic compounds collected by the following aromatic-compounds recovery process, may be caused. On the other hand, big equipment and energy are needed and it is not economical to use 1 ppm or less. the operating pressure power of a distillation column -- decompression, ordinary pressure, and application of pressure -- it being feeding into a hydrogen chloride recovering process the liquid which kept this pressure higher than the pressure of a hydrogen chloride purification process, and was obtained by the chlorination purification process using a pump, and, although any may be sufficient, It can supply and refine to a hydrogen chloride purification process, without compressing the hydrogen chloride gas obtained from the overhead of the distillation column. Under the present circumstances, the gas obtained from the overhead can once be cooled by a heat exchanger, some the aromatic compounds and chlorinated aromatics which are contained in gas can be made to be able to condense, and a tower can also be refluxed.

#### **0018**

In this invention, the fraction which is mainly concerned with the aromatic compounds and chlorinated aromatics which were obtained by the above-mentioned hydrogen chloride recovering process is supplied to the following aromatic-compounds recovery process.

Aromatic-compounds recovery process: The process of separating the fraction with which it is mainly concerned, and collecting aromatic compounds from the fraction which is mainly concerned with aromatic compounds and chlorination aromatic series

#### **0019**

It is as follows when the example which carries out an aromatic-compounds recovery process is given.

## 0020

The fraction which is mainly concerned with the aromatic compounds and chlorinated aromatics which were obtained by the middle part of the distillation column which equips a crowning with a capacitor and equips a pars basilaris ossis occipitalis with a reboiler by the hydrogen chloride recovering process is supplied. The fraction which is rich in aromatic compounds is obtained from the overhead by liquefying the steam obtained from the overhead by heating with a reboiler by a capacitor, and refluxing the part in the crowning of a distillation column. Although only the fraction which is mainly concerned with the aromatic compounds and chlorinated aromatics which were obtained by the hydrogen chloride recovering process may be supplied to a distillation column, For example, the liquid which consists of chlorination aromatic series which is obtained from a chlorination process, and which was generated at unreacted aromatic compounds and reaction can be supplied collectively, and aromatic compounds can also be collected. In this invention, the fraction which is mainly concerned with the aromatic compounds obtained by the aromatic-compounds recovery process can be supplied to a chlorination process. The recycling use of the aromatic compounds can be carried out useful by this.

## 0021

In this invention, the gas which is mainly concerned with the hydrogen chloride obtained by the hydrogen chloride purification process can be supplied to the following oxidation process. By this, hydrogen chloride can be changed into chlorine and this chlorine can be used effectively.

Oxidation process: The process of making hydrogen chloride reacting to oxygen and obtaining chlorine

## 0022

An oxidation process is a process of making the hydrogen chloride obtained at the chlorination process reacting to oxygen, and obtaining chlorine. About the method to which hydrogen chloride and oxygen are made to react, there is no restriction in particular and a publicly known method can be used. It is as follows when the example of a concrete method is shown. The mole ratios (hydrogen chloride/oxygen) of hydrogen chloride and oxygen are 0.5-2, 200-500 \*\* of reaction temperature is 200-380 \*\* preferably, reaction pressure is 0.1 - 5MPa, and superficial velocity is 0.7 - 10 m/s. As a reactor, a fixed bed reactor, a fluid bed reactor, and a moving bed reactor can be used. A chromium oxide catalyst and a ruthenium oxide catalyst can be used for a reaction as a catalyst.

## 0023

At least some chlorine obtained by the oxidation process is recycled to a chlorination process, and it is used effectively as a raw material of a chlorination process.

## 0024

By adopting the above-mentioned mode, chlorinated aromatic hydrocarbon (for example, chlorobenzene) as the last object can be efficiently manufactured from aromatic hydrocarbon (for example, benzene) and chlorine.

## Example

## 0025

Next, an example explains this invention.

### Example 1

This invention can be carried out the optimal by the flow of drawing 1, and the material balance of Table 1.

Chlorine was made to react to benzene and chlorobenzene and hydrogen chloride were obtained. Reaction temperature set 117 \*\* and reaction pressure to 0.6MPa here, and the mole ratio of the benzene/chlorine supplied from the outside could be 8/1. The benzene which cooled the benzene by which it was generated at the reaction, and the gas containing hydrogen chloride to 30 \*\*, and was condensed was returned, and took out the mixed gas (fluid number-6) which contains benzene and hydrogen chloride by 0.03/1. **Chlorination process**

Above-mentioned mixed gas and chlorobenzene were contacted in the bottom pressure 0.55MPa and whose temperature are -35 \*\*, and the solution (fluid number-8) which serves as hydrogen chloride gas (fluid number - nine) by which the concentration of benzene and chlorobenzene was reduced from hydrogen chloride hydrogen, benzene, and chlorobenzene was obtained. **Hydrogen chloride purification process**

The hydrogen chloride obtained by the chlorinating reaction in the solution obtained by the

hydrogen chloride purification process, The hydrogen chloride which heats under pressure 0.6MPa and is dissolving into liquid with the liquid containing benzene and chlorobenzene is made to diffuse, The mixed liquor (fluid number-13) of benzene, the benzene in which the molar concentration of hydrogen chloride was reduced from the bottom in the mixed gas (fluid number - 14) which contains hydrogen chloride by 0.02/1, and chlorobenzene was obtained from the overhead. **Hydrogen chloride recovering process**

The mixed gas obtained by the hydrogen chloride recovering process was returned and re-refined to the hydrogen chloride purification process. The usual distillation separated into benzene (fluid number-16) and chlorobenzene (fluid number - 15) the mixed liquor of the benzene obtained from the hydrogen chloride recovering process, and chlorobenzene. **Aromatic-compounds recovery process**

The benzene separated and collected by the aromatic-compounds recovery process was recycled as a raw material of a chlorination process. The hydrogen chloride gas obtained by the hydrogen chloride purification process was made to react to oxygen, and was changed into chlorine and water. **Oxidation process**

The obtained chlorine (fluid number-12) was recycled as a raw material of a chlorination process.

**0026**

**Table 1**

✕ ID=000003

### **Brief Description of the Drawings**

**0027**

**Drawing 1** It is an example of the flow which carries out this invention.

### **Description of Notations**

**0028**

A: Chlorination process

B: Hydrogen chloride purification process

C, F: Pump

D: Hydrogen chloride recovering process

E: Aromatic-compounds recovery process

G: Oxidation process

---

### **Brief Description of the Drawings**

**0027**

**Drawing 1** It is an example of the flow which carries out this invention.

---

## Drawing 1

x ID=000004

